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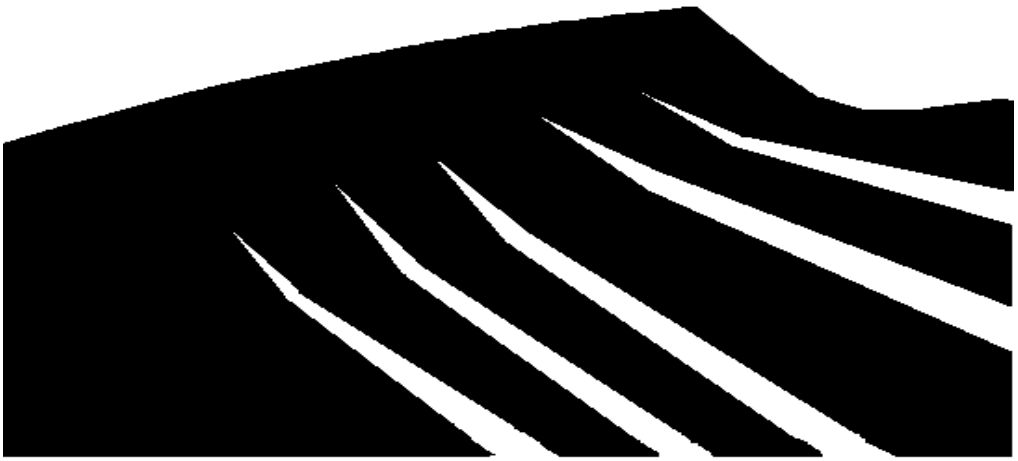
PREPARATION OF STANDARDS FOR TRACER CONCENTRATION MEASUREMENT

LOS ALAMOS

QUALITY PROGRAM



APPROVAL FOR RELEASE	
P. W. REIMUS - PREPARER Signature on file	DATE Date on file
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Los Alamos
Yucca Mountain Site Characterization Project

HISTORY OF REVISION

REVISION NO.	EFFECTIVE DATE	PAGES REVISED	REASON FOR CHANGE
R0	01/18/96	N/A	Initial procedure.
R1	05/10/96	5, 8, & 9	To address M&TE calibration and training requirements.
R2	01/21/97	All	Minor non-substantive editorial changes

PREPARATION OF STANDARDS FOR TRACER CONCENTRATION MEASUREMENTS

1.0 PURPOSE

This detailed procedure (DP) describes the preparation of standards used to determine concentrations of tracers in ground water samples collected in tracer transport experiments. The DP is intended to be used in cases where it may not be practical or desirable to use NIST-traceable standards as concentration standards. For example, because of the large quantities of tracers required in field tracer transport experiments, it is often much more cost-effective to use lower purity tracers than analytical-grade tracers. In these cases, it is often desirable to prepare standards directly from the tracer stock rather than from NIST-traceable standards because the stock will inherently include impurities that were included in the tracer tests. Also, it is often desirable to prepare standard solutions directly in the ground water from the field site (or the lab experiment) because standards prepared in this way will inherently contain any interferences or background concentrations that will be present in the samples to be analyzed. It is also recognized that when tracer transport experiments are interpreted, it is almost never necessary to know the absolute concentration of tracers in the samples collected, but rather it is more important to know tracer concentrations relative to the amount of tracer that was injected into the experiment. Thus, the standards should logically be prepared using the tracer stock used in the tracer experiment rather than NIST-traceable or other high-quality standard that is intended to allow absolute concentration measurement. This DP is intended to support the C-Wells Reactive Tracer Studies for the Yucca Mountain Project (YMP), although it could also be used for other YMP studies.

2.0 SCOPE

This DP applies to the preparation of standards for any analytical procedure involving tracer concentration measurements in which the measurements technique relies on relating the measurement of an unknown to the measurements of a standard or a set of standards. The analytical measurements themselves will be covered by DP's that address the operation of the analytical instrumentation. This procedure also applies to the handling and storage of standards.

3.0 REFERENCES

LANL-YMP-QP-02.7, Personnel Training
LANL-YMP-QP-03.5, Documenting Scientific Investigations
LANL-YMP-QP-08.1, Identification and Control of Samples
LANL-YMP-QP-12.3, Control of Measuring and Test Equipment and Standards
LANL-YMP-QP-17.6, Records Management

4.0 DEFINITIONS

4.1 Standard

For the purposes of this DP, a standard is a solution containing an accurately known concentration of a tracer or a set of tracers that have been used in a tracer transport experiment. The standard (or, more generally, a series of standards) will be used to calibrate or obtain a calibration curve for analytical instrumentation that is used to determine the concentration of tracers in samples collected from a tracer experiment.

5.0 RESPONSIBILITIES

The following personnel are responsible for the activities identified in Section 6.0 of this procedure.

- Principal Investigator (PI)
- Procedure User

6.0 PROCEDURE

The use of this procedure must be controlled as follows:

- If this procedure cannot be implemented as written, YMP personnel should notify appropriate supervision. If it is determined that a portion of the work cannot be accomplished as described in this DP, or would result in an undesirable situation, that portion of the work will be stopped and not resumed until this procedure is modified or replaced by a new document or until the current work practice is documented in accordance with QP-03.5, subsection 6.1.6.

- Employees may use copies of this procedure printed from the controlled document electronic file; however, employees are responsible for assuring that the correct revision of this procedure is used.

- When this procedure becomes obsolete or superseded, it must be destroyed or marked “superseded” to ensure that this document is not used to perform work.

6.1 Principle

Most analytical determinations of species concentrations in solutions involve measurements that must be related to measurements of known concentrations of the species in solution (i.e., the measurements are relative, not absolute, and therefore they must be compared to measurements of known concentrations). The solutions containing the known concentrations are called standards.

6.2 Equipment and Hardware/Software

The equipment required for this DP includes balances for accurate determination of masses of reagents and Class A volumetric glassware, including pipettes and volumetric flasks. In certain situations, other equipment, such as stirrers and temperature measurement and/or control devices may also be required. Any balances that are used should be calibrated and controlled in accordance with QP-12.3, or they should be calibrated at each use using a weight set that is traceable to NIST standards. When calibrating at each use, the calibrations should be documented in a field or laboratory notebook in accordance with QP-03.5 (see QP-12.3).

6.2.1 Equipment Malfunctions

Malfunctions of any of the equipment described above will be either immediately obvious to the user or will result in the inability to conduct the procedures described in this DP.

6.2.2 Safety Considerations

Safety considerations will depend on the chemical nature of the constituents in the solutions being prepared. Material safety data sheets (MSDSs) should be consulted to determine whether special protective clothing and/or eye protection are required. Hazardous chemical wastes should be properly disposed of.

6.2.3 Special Handling

Handling of all equipment associated with this DP should be done in accordance with manufacturer's or vendor's guidelines. Special handling of equipment or hardware should be considered on a case-by-case basis as the need arises. Any special handling should be documented in a laboratory notebook.

6.3 Preparatory Verification

The DP user should ensure that all glassware used to prepare standards is Class A volumetric, and that any balances used are calibrated and controlled in accordance with QP-12.3. Alternatively, balances can be calibrated at each use using a weight set that is traceable to NIST standards. In this case, the calibrations should be documented in a field or laboratory notebook in accordance with QP-03.5 (see QP-12.3).

6.3.1 Hold Points

(N/A)

6.3.2 Calibration

In general, calibration of equipment other than balances is not required when implementing this DP. However, Class A volumetric glassware must be used when preparing solutions.

6.3.3 Environmental Conditions

When standards are prepared, good laboratory practices should be followed to maintain a clean laboratory environment to avoid contamination of the standards, and the standards should be prepared at room temperature to minimize volumetric errors (unless the volumetric glassware requires another temperature).

6.4 Control of Samples (Standards)

Standard solutions are to be identified and controlled as “samples” in accordance with QP-08.1. Standards can be stored in any airtight container that does not compromise them by introducing contaminants that could interfere with the analyses. Glass and plastic bottles are generally acceptable, although the choice of lab ware should consider the potential sorption of tracers to container walls. If sorption is unavoidable, special procedures should be followed to desorb the tracers prior to analyses (e.g., acidification). These procedures should be documented in a laboratory notebook. If there is any question about sorption to container walls, batch sorption experiments should be conducted (and documented in a laboratory notebook) using the tracers and lab ware in question. Standard storage should consider the stability of the tracers. For example, if a UV light sensitive tracer is used (e.g., a fluorescent dye such as fluorescein), the samples should be stored in darkness or in UV opaque bottles. Standards should generally be analyzed at the same temperature as the unknowns.

6.5 Implementing Procedure

Preparation of standards for tracer concentration measurements involves the following steps:

6.5.1 Starting Material for Standards Preparation

A portion of the tracer material that was actually used in the tracer experiment (generally a powder, but possibly a solution) should be held back when the tracer experiment is conducted. A small amount of this material should be accurately weighed using a properly calibrated balance. If the tracer material is a solution, the weight can be used to determine a volume (assuming that the density is known). Alternatively, for solutions, it may be necessary to use Class A volumetric glassware to dispense a known volume of the solution. In some cases, it may be desirable to simply use an aliquot of the tracer mixture that was prepared for injection into the tracer transport experiment (assuming that

the solution was well-mixed prior to collection of the aliquot, and that the weight or volume of tracer in the mixture and the total volume of the mixture are accurately known). The DP user should record in a field or laboratory notebook all weight readings, volume measurements, and / or any other steps taken to accurately determine the mass or volume of tracer material that is used for standards preparation. This material will be referred to as the "starting material" in the procedure described below. The unique identifier of any Measuring and Test Equipment (e.g., balances) should also be recorded.

6.5.2 Standard Preparation

Standards preparation generally involves preparing a series of dilutions of the starting tracer material (see subsection 6.5.1 for description of "starting material") to obtain a set of standards that span the expected range of concentrations in the unknowns. If the starting material is a powder or other solid, this material should be dissolved or mixed in a small volume of tracer-free ground water collected from the tracer test site or in whatever water was used in the experiment. If ground water or water used in the experiment is not available, the PI should make a determination of what type of solvent should be used to prepare standards (deionized water will generally be the logical choice). The mixture should then be diluted in the ground water (or other solvent) to a known volume using Class A volumetric glassware. Subsequent dilutions to prepare lower concentration standards can be done in series by pipetting a known volume of each successive standard prepared (starting with the first, most concentrated one) into a volumetric flask (all Class A glassware) and then diluting to the mark with the appropriate tracer-free solvent. This procedure, known as "serial dilution", is a widely-recognized and accepted method of preparing standards for a wide range of chemical analysis techniques. The nominal accuracy of the glassware used ($\pm$ X%) should be documented in a field or laboratory notebook; or, conversely, accuracy can be determined / documented by weighing fluid quantities delivered (pipettes, micro pipettes) or contained (flasks) using a calibrated balance. The DP user should record in a field or laboratory notebook all actions taken to prepare standards, including the weights or volumes used / measured in each step of the procedure, the identity of the glassware used, the ambient temperature in the laboratory (if applicable), and any unusual occurrences or situations that could result in errors in the standards concentrations.

NOTE: The PI may determine that it is desirable to prepare a separate set of standards in deionized water or in some other solution that does not contain species that may be present in the ground water. This might be done, for instance, to quantify the background concentration of tracers in the tracer-free ground water or perhaps to determine / verify the purity of the starting material. In such instances, the PI or DP user should clearly document the purpose for preparing the separate set of

standards as well as the solution to be used for the standards, and the standards should be prepared in the same way as described above except that the appropriate solution should be used in place of ground water. Care should be taken to document traceability from the “serial dilution” to the parent material.

6.6 Data Acquisition and Reduction

With the exception of weight readings on balances and calculations to determine concentrations of standards based on weights and volumes, this DP does not involve data acquisition or reduction. Data acquisition and reduction associated with analyses of tracers will be covered by DP’s that control the operation of the appropriate analytical instrumentation. Sources of error and uncertainty associated with this DP are discussed in subsection 6.7.

The criteria for acceptance of prepared standards are the (1) all of the steps taken to prepare the standards are properly documented as prescribed in subsection 6.5, (2) any balances used were properly calibrated and were either listed on the YMP Measuring and Test Equipment (M&TE) List, or were calibrated at each use using a weight set traceable to NIST standards, and (3) Class A volumetric glassware was used for all dilutions involved in standards preparation. The PI ultimately reviews all data and records associated with standards preparation and determines the acceptability of the standards and the tracer concentration measurements that are conducted using the standards. The PI may reject standards if (1) it appears that the standards do not follow expected trends when they are analyzed with analytical equipment (e.g., standards that are supposed to have a factor of 10 difference in concentration give responses that suggest only a factor of 5 difference), or (2) the tracer concentration measurements indicate tracer behavior that is clearly erroneous or impossible (e.g., 120% recovery of tracer in a tracer experiment). The identity of rejected standards and the basis for rejection are recorded in a field or laboratory notebook.

6.7 Potential Sources of Error and Uncertainty

Potential sources of error and uncertainty associated with the implementation of this DP include (but are not necessarily limited to) the following:

- Inaccurate weight or volume measurements,
- Inadvertent use of the wrong volumetric glassware,
- Use of dirty glassware that contains contamination or significant levels of the constituents that interfere with the analyses of the target tracer,
- Preparing standards at a significantly different temperature than the volumetric glassware is intended for, and

- Improper storage of standards such that they degrade or are otherwise compromised.

If a problem arises during standards preparation, handling, or storage that can be considered a potential source of error or uncertainty when conducting tracer analyses, the PI or DP user should document it in a field or laboratory notebook. The PI or DP user can then later assess the impact of the source of error or uncertainty when analyzing the measured tracer concentrations.

7.0 RECORDS

Records generated as a result of this DP are entries in field or laboratory notebooks or attachments to such notebooks. The documentation should consist of any applicable items identified in Section 6.0. Notebooks should be kept in accordance with QP-03.5.

All records should be submitted to the Records Processing center in accordance with QP-17.6.

8.0 ACCEPTANCE CRITERIA

Proper completion and submittal of the records listed in Section 7.0 constitutes the acceptance criteria for this procedure.

9.0 TRAINING REQUIREMENTS

Read-Only training is required for this DP. Training is documented in accordance with QP-02.7.

10. ATTACHMENTS

(N / A)